Large Tapered Crystal (LTC) Growth Method: A New Single Crystal Silicon Carbide Bulk Growth Technique

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ANFF-AFOSR Review

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National Aeronautics and Space Administration

What is Silicon Carbide (SiC)?

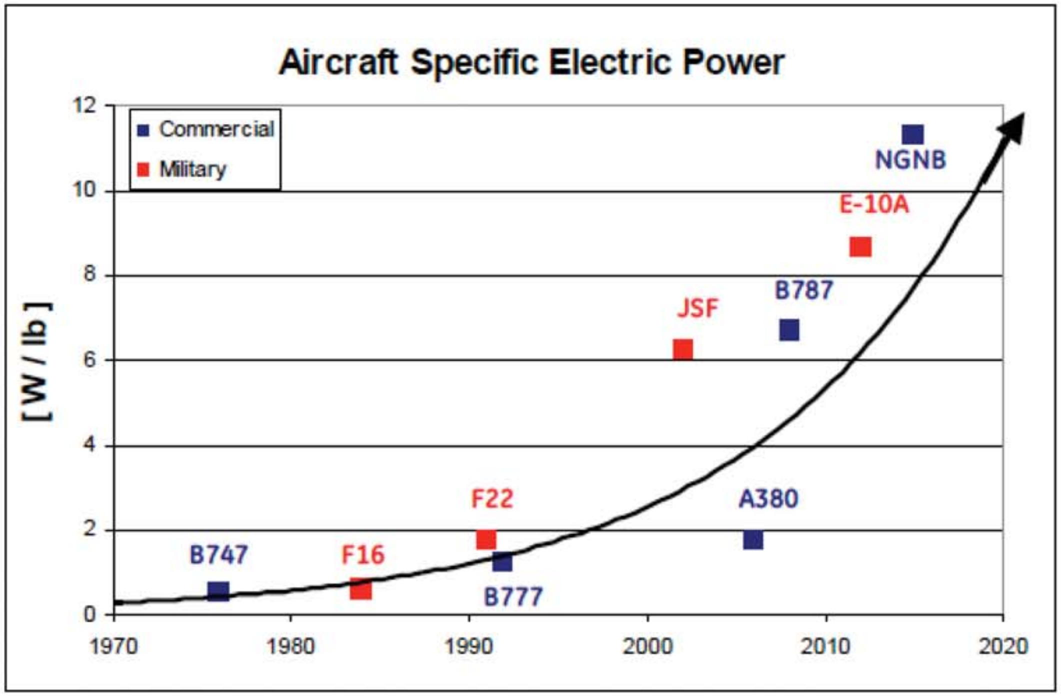
- Si and C sp^3 bonded (much like diamond)
- 212 polytypes (crystallographic structures)
- Chemically inert
- Wide band gap semiconductor

David Monniaux , Silicon carbide (SiC) monocrystal from the LMGP (Minatec) lab in Grenoble, France

Applications

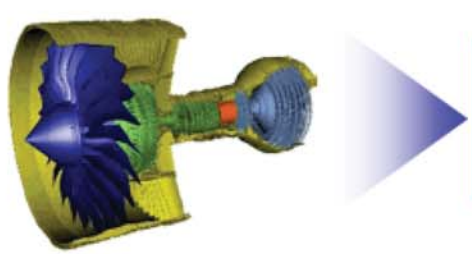
- Abrasives
- Structural
- Electronics
    - Power systems
    - High temperature environments
    - Harsh environment

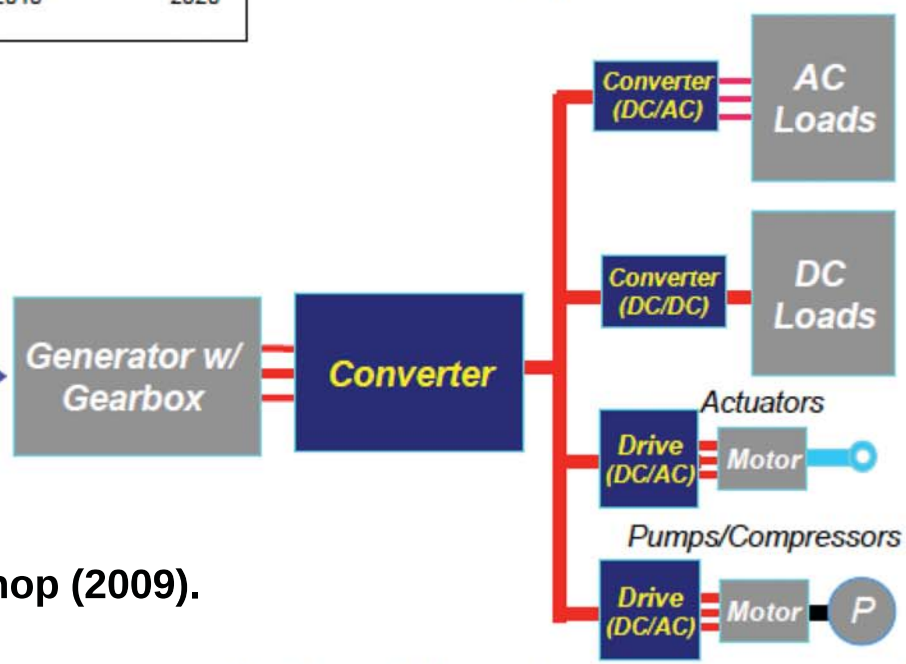
SiC for Aerospace Applications



Aviation Industry Trends:

- Demand for higher energy efficiency, lower GHG emissions
- Replacing hydraulic and pneumatic systems with more-electric architecture
- Power conversion efficiency and size critical for more-electric architecture
- Flight profiles of new military planes limited by power electronics thermals

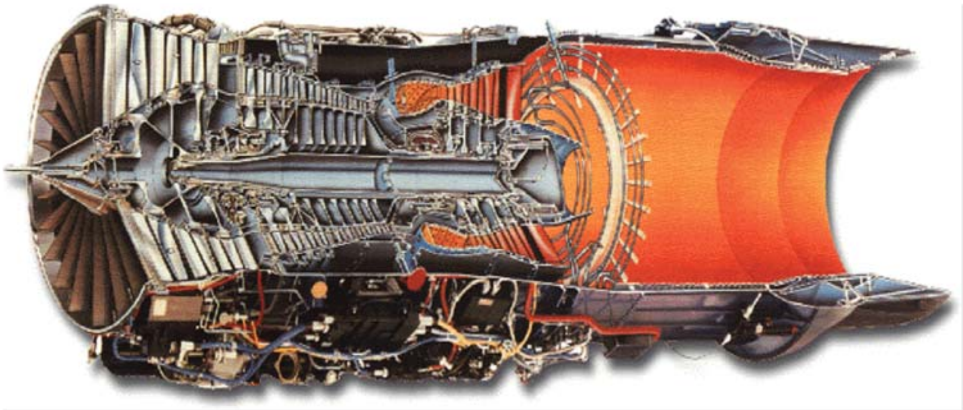




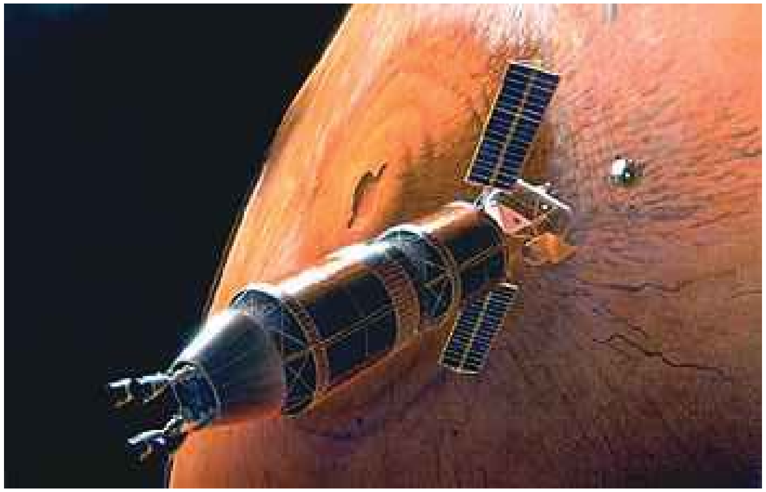
L. Stevanovic, GE, EETWBGPE Workshop (2009).

SiC Electronics Benefits to NASA Missions

Intelligent Propulsion Systems

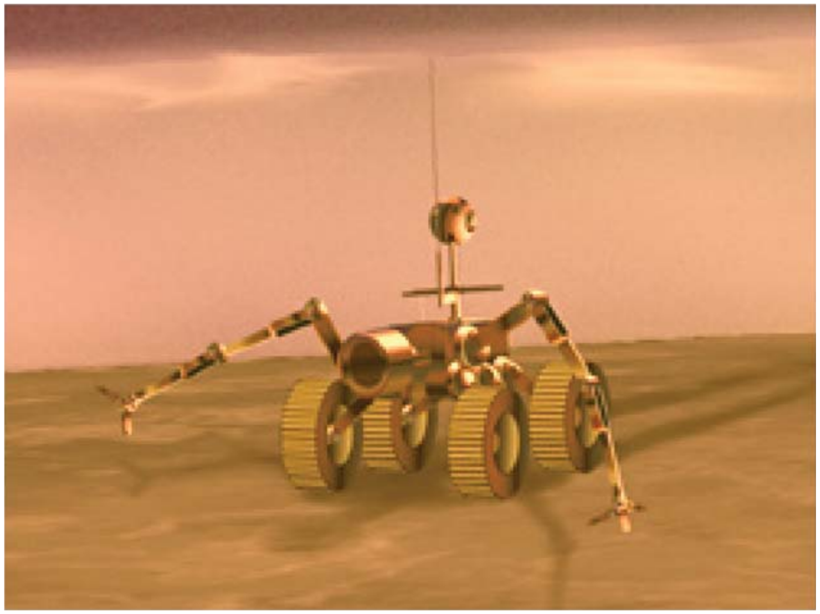


Space Exploration Vision PMAD



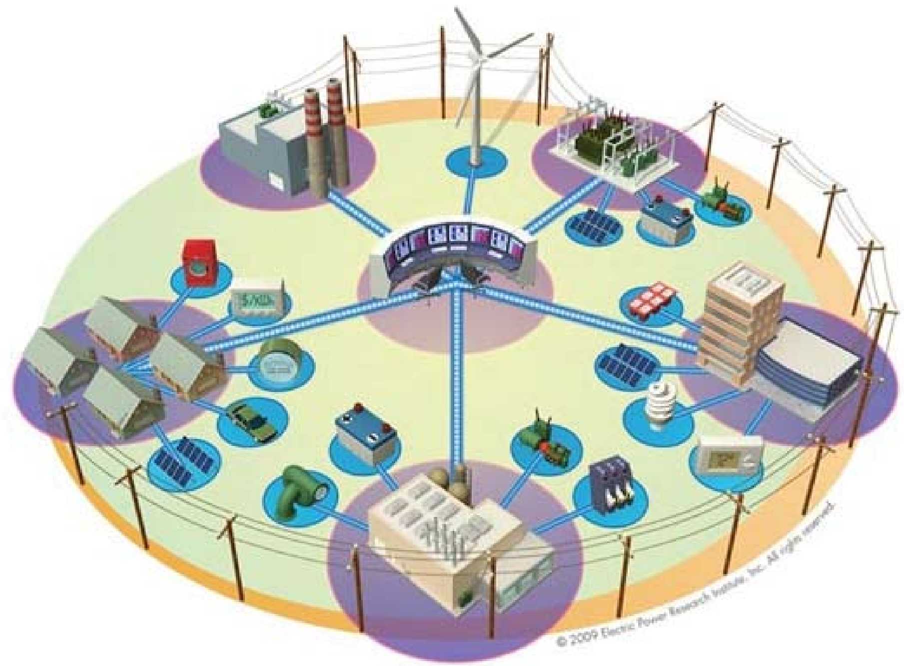
More Electric + Distributed Control Aircraft

Venus Exploration



All combinations of <u>high temperature and/or high power</u> applications!

DOE Applications



http://www.smartgrid.epri.com/Demo.aspx

- Smart Grid
    - Ability to network many sources and sinks
    - Need to minimize losses in complex system
- Electric and hybrid electric vehicles
    - Minimize weight and size of converters
    - Minimize or eliminate cooling requirements

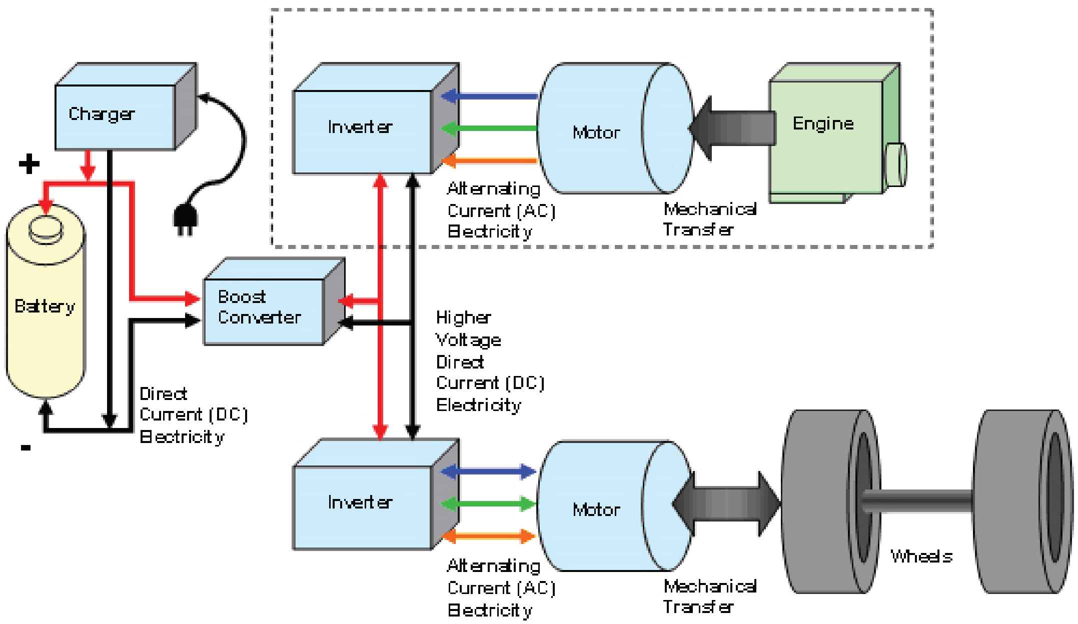
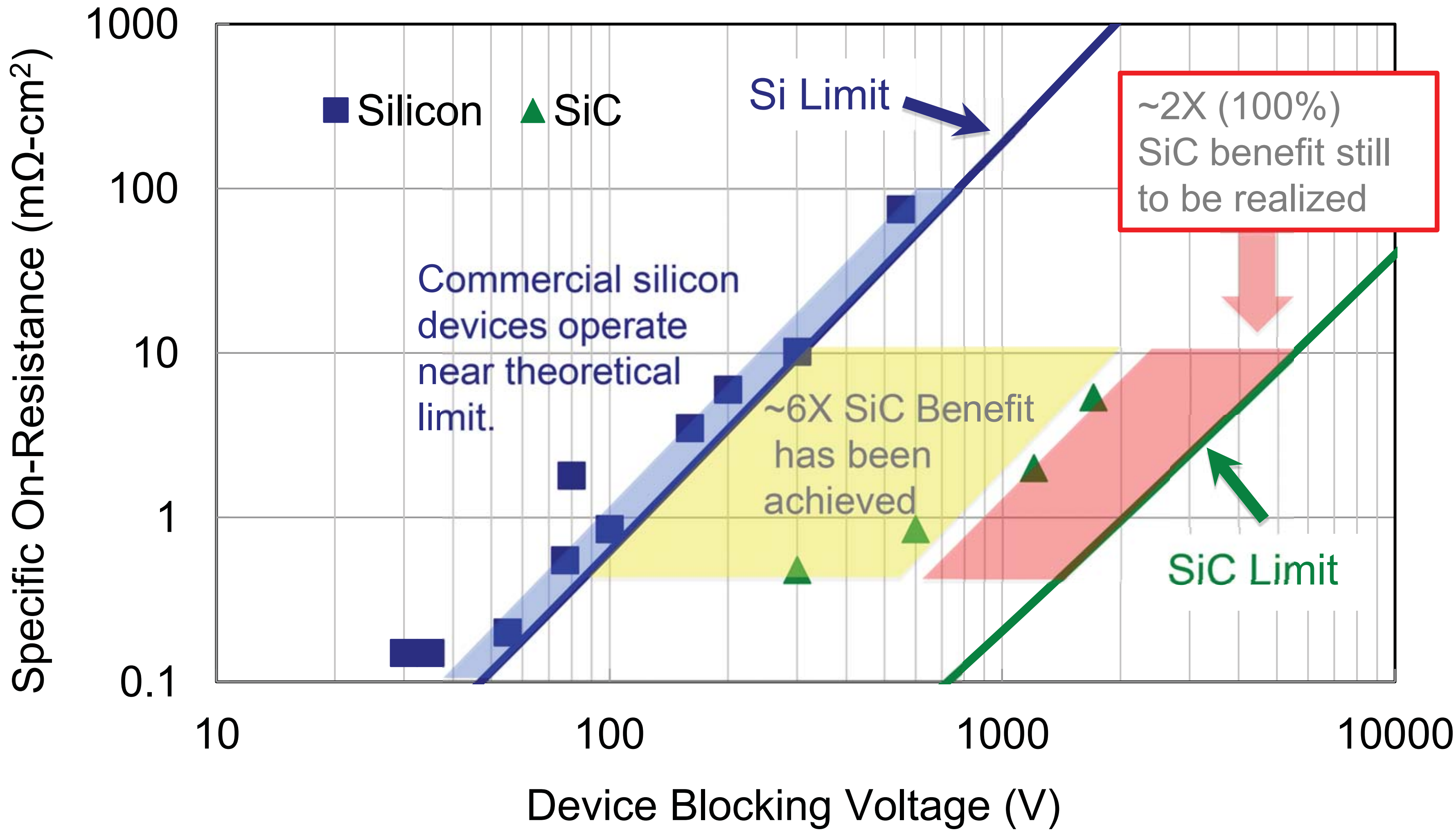


Diagram of a power electronics and electrical machines in a plug-in hybrid electric vehicle (PHEV). http://www1.eere.energy.gov/vehiclesandfuels/technologies/electronics/index.html

Unipolar Power Device Comparison

(Volume Production Commercial Devices)

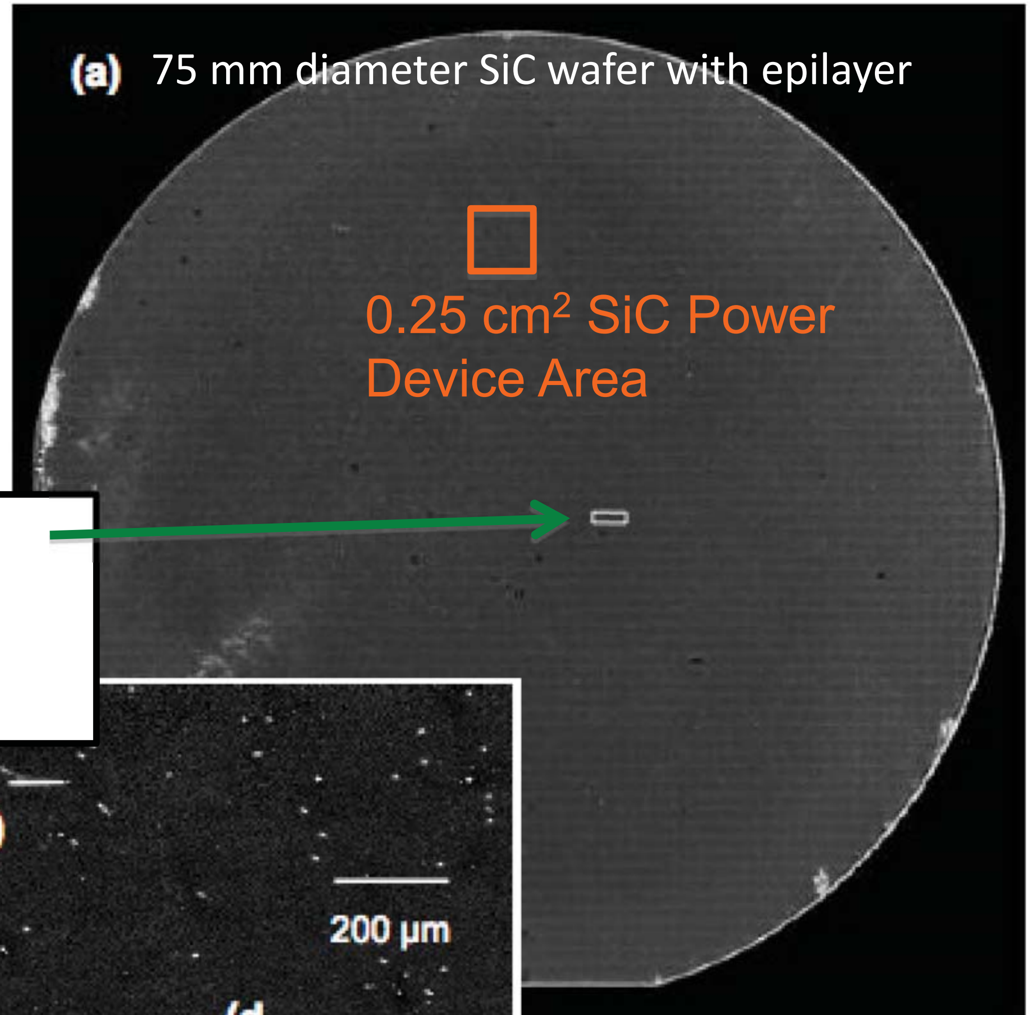
SiC devices are ~2X voltage or current-density **de-rated** from theoretical material performance.



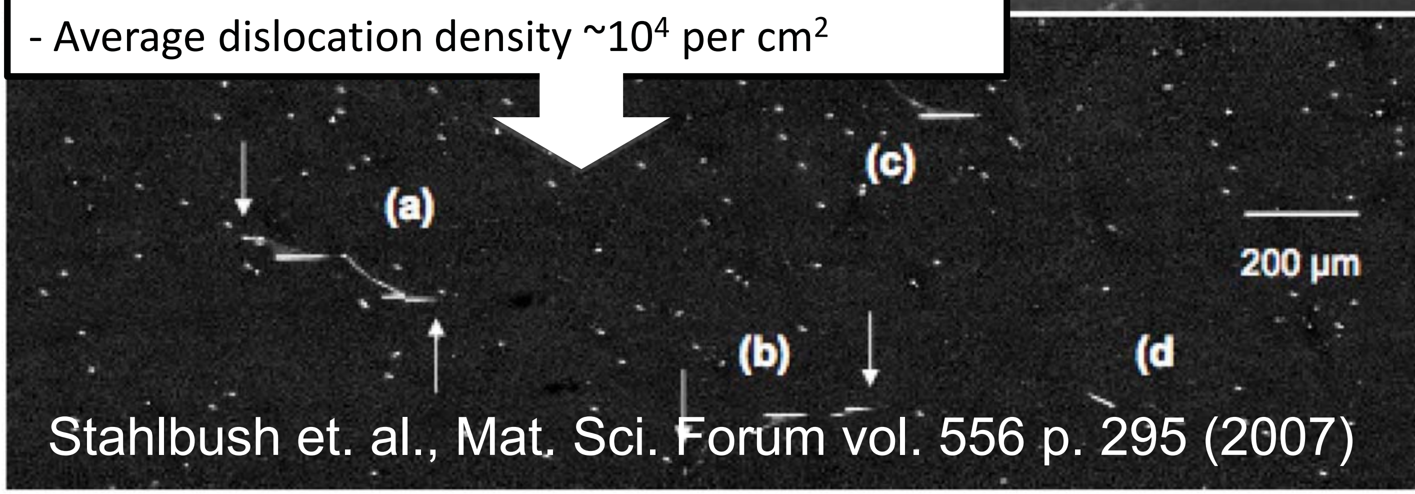
Above comparison does NOT take yield, cost, other relevant metrics into account.

SiC Wafer Material Defects

Over the past decade there have been numerous studies (including NASA GRC) linking degraded SiC power device performance, yield, and reliability to the presence of defects in the SiC wafer crystal.



Magnified view small area in middle of wafer imaged by Ultra-Violet Photoluminescence
- Each white dot or line is a dislocation defect!
- Average dislocation density ~10^4 per cm^2



Stahlbush et. al., Mat. Sci. Forum vol. 556 p. 295 (2007)

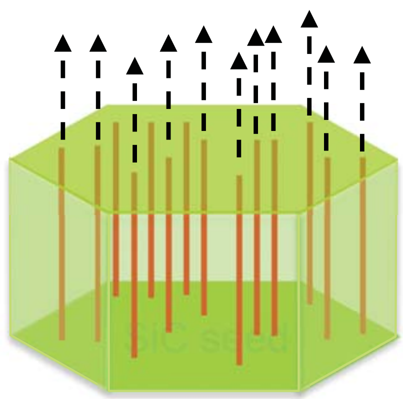
Two-fold defect-induced SiC device over-design roughly translates into corresponding energy loss and/or power circuit size increase trade-off.

Description of Technology/Approach

Large Tapered Crystal (LTC) SiC Growth

Present SiC Growth Process

(Vapor transport)



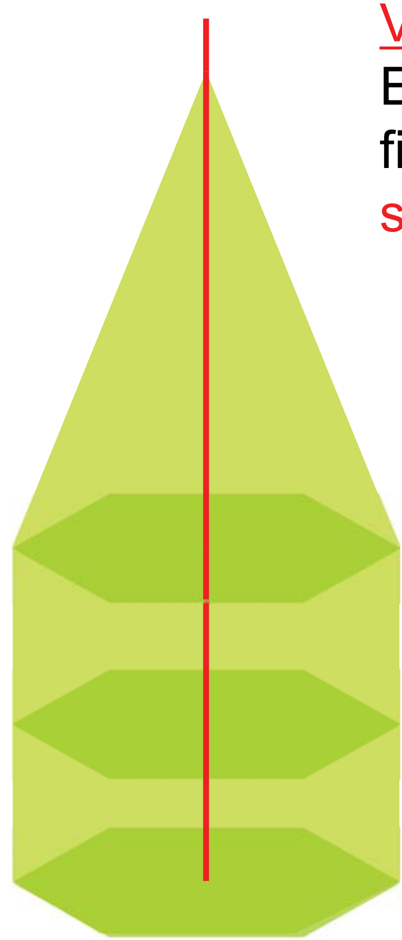
Vertical (c-axis) growth proceeds from top surface of large-area seed via thousands of screw dislocations.(i.e., dislocation-mediated growth!)

Crystal grown at T > 2200 ° C
High thermal gradient & stress.

Limited crystal thickness.

Proposed LTC Growth Process

(US Patent 7,449,065 OAI, Sest, NASA)



Vertical Growth Process:
Elongate small-diameter fiber seed grown from single SiC dislocation.

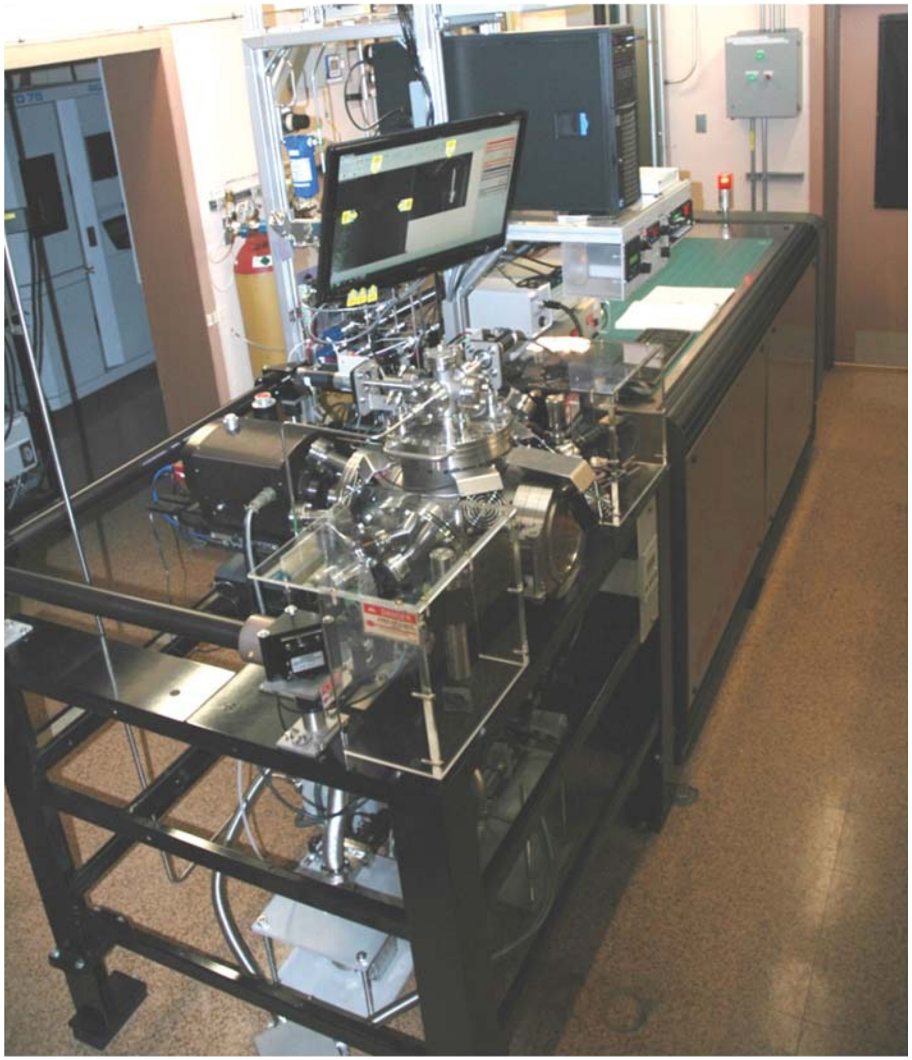
Lateral Growth Process:
CVD grow to enlarge fiber sidewalls into large boule.
- 1600 ° C, lower stress
- Only 1 dislocation

Lateral & vertical growth are simultaneous & continuous (creates tapered shape).

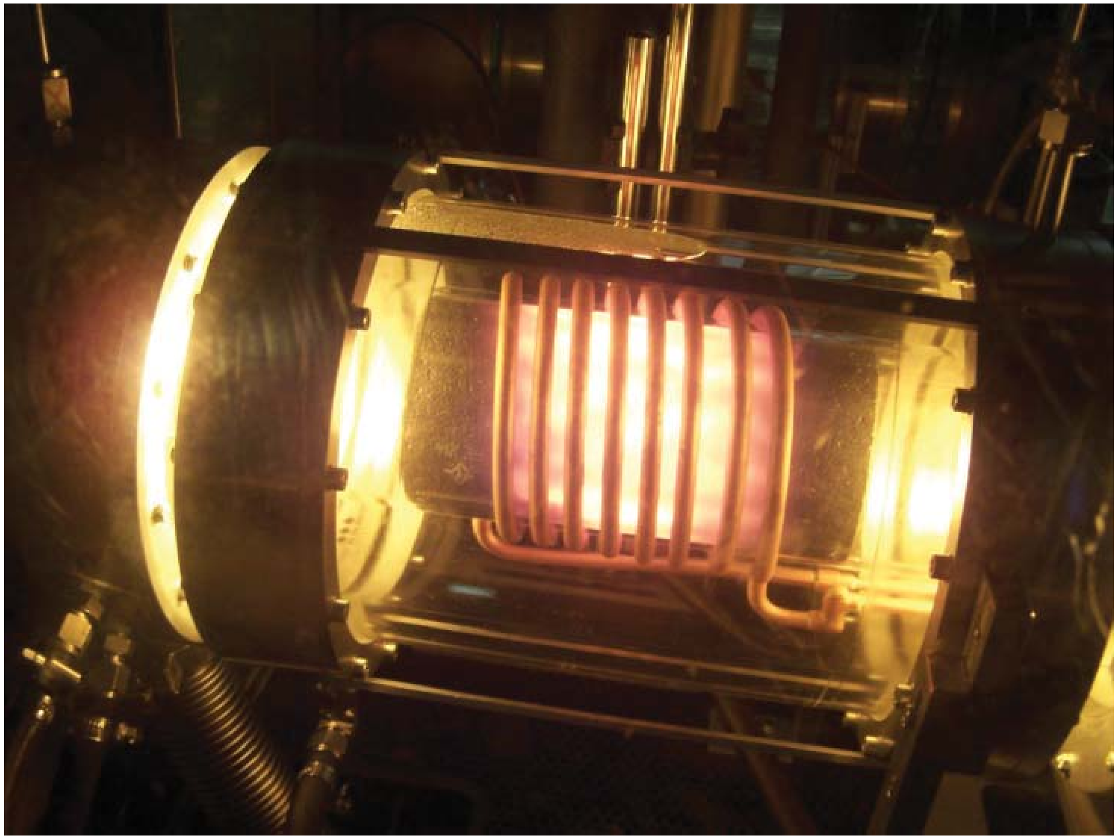
Radically change the SiC growth process geometry to enable full SiC benefit to power systems.

LTC Development : Two track approach

SiC fiber growth by Solvent-Laser Heated Floating Zone (Solvent-LHFZ)



Lateral growth by Chemical Vapor Deposition (CVD)

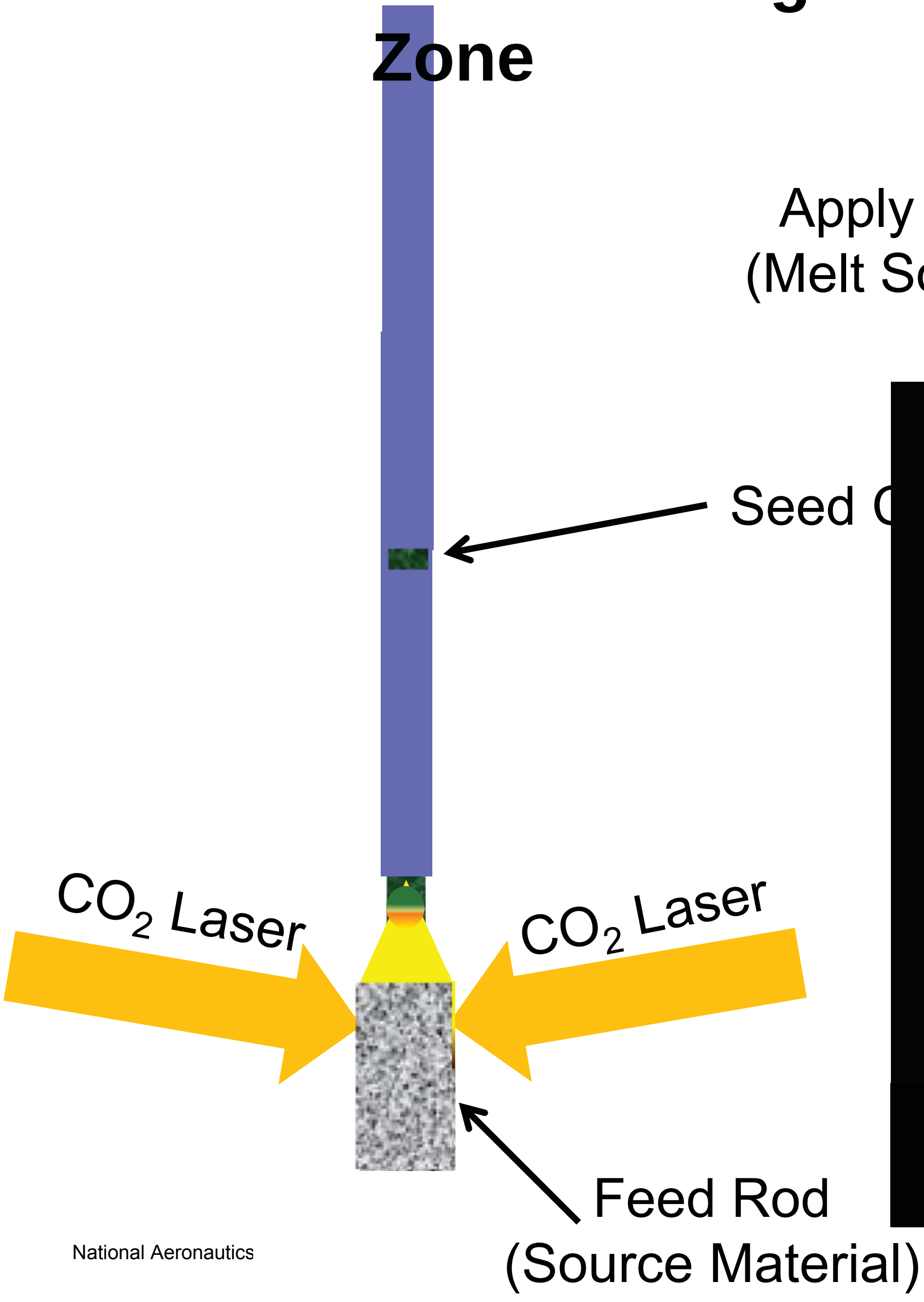


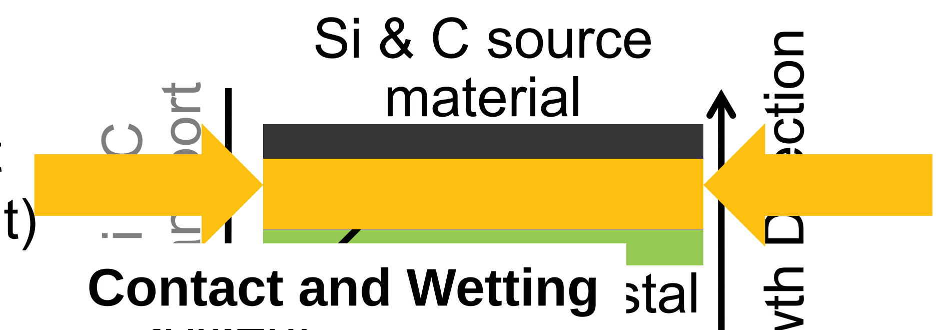
Solvent-LHFZ Technique

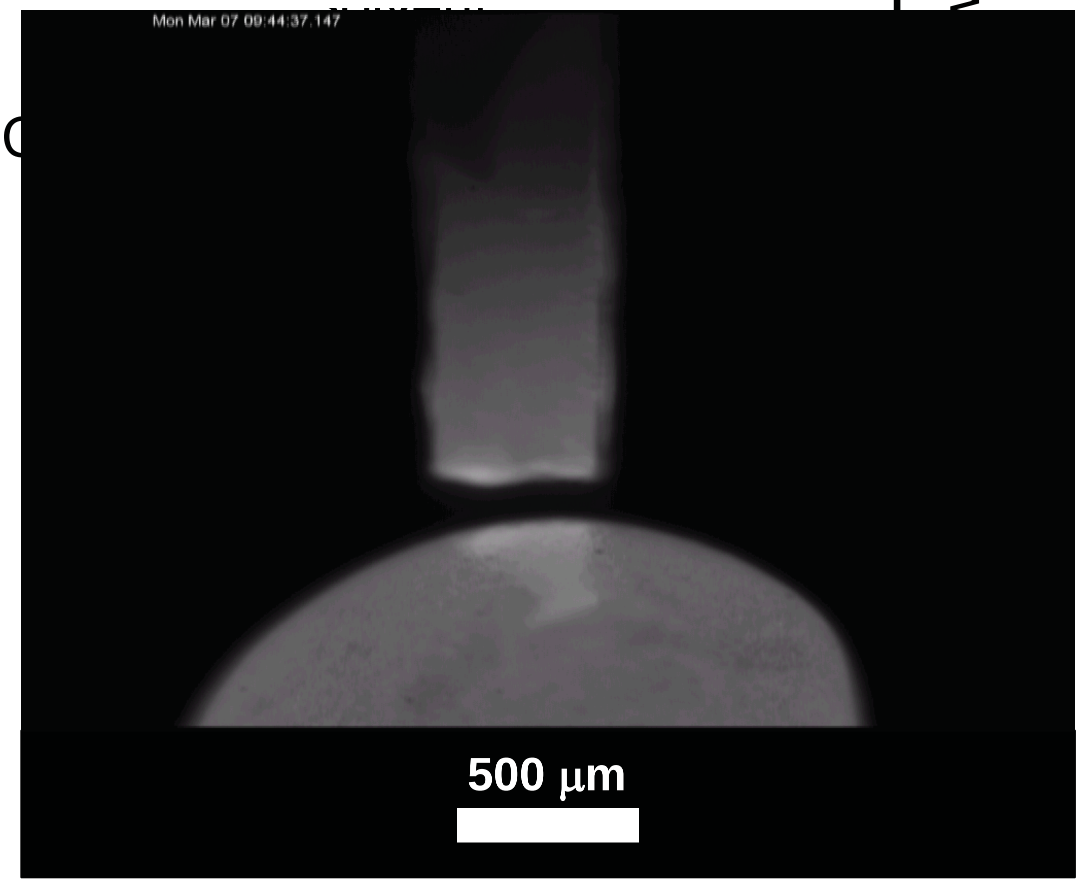
Laser Heated Floating Zone

+

Solvent Growth Method

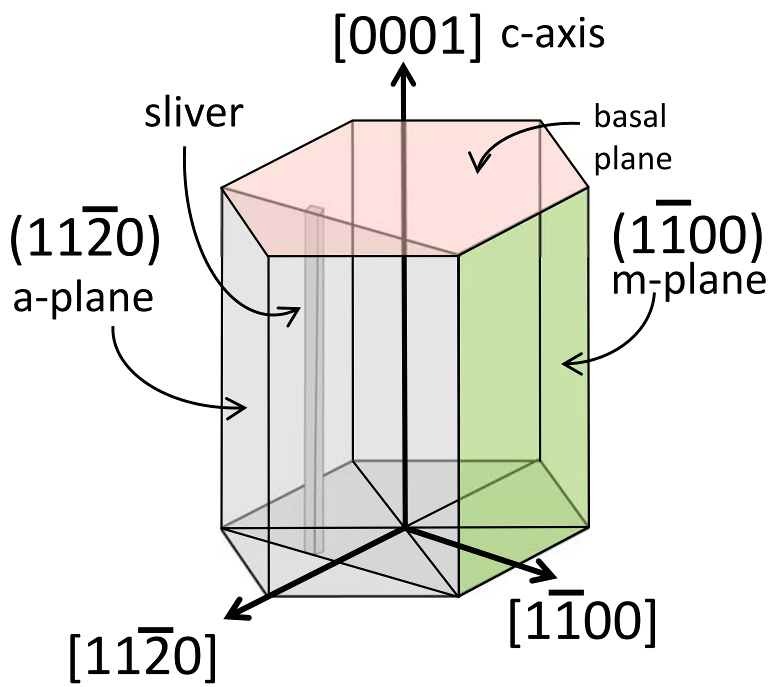


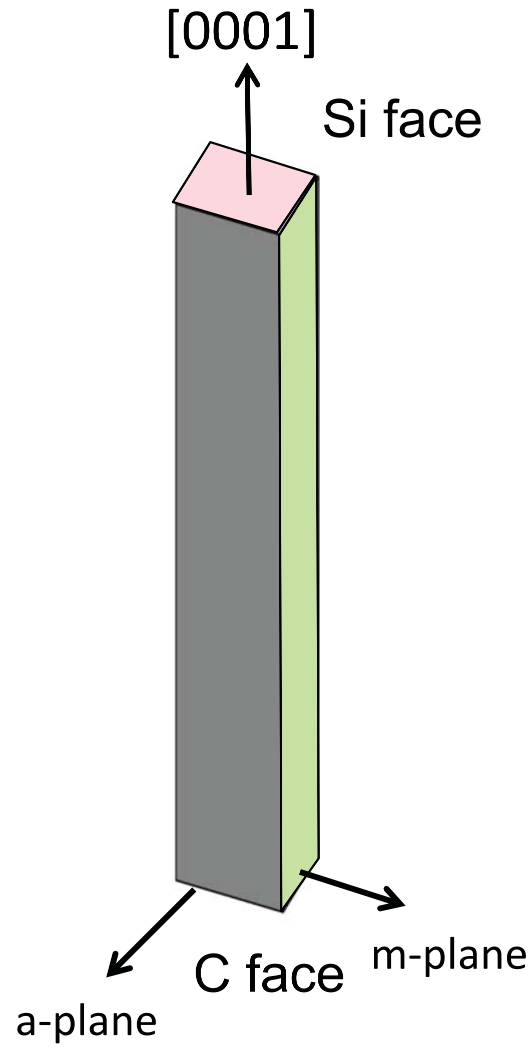




Seed Crystals

4H-SiC





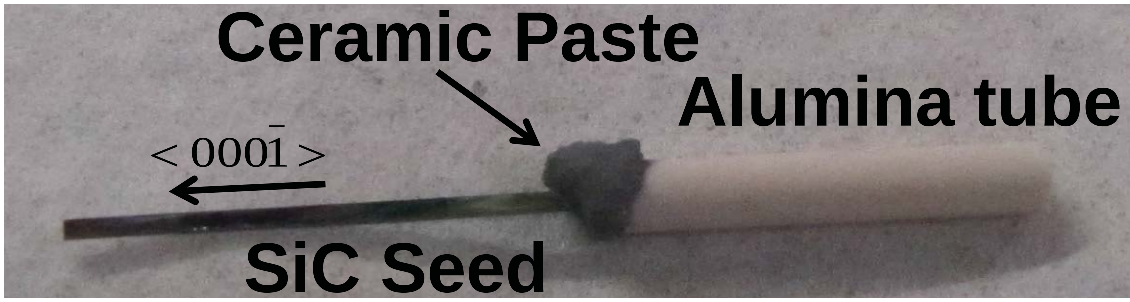
Seed Crystals

Growth face

- 4H-SiC C-face (0-10º off axis)
- ~500 μm X ~450 μm

Mounting

- Seed ~1.5 cm long
- Ceramic pasted into an alumina tube
- After curing seed crystals cleaned
    - $HCl:HNO_3$ (2:1)
    - HF



Source Material / Feed Rod

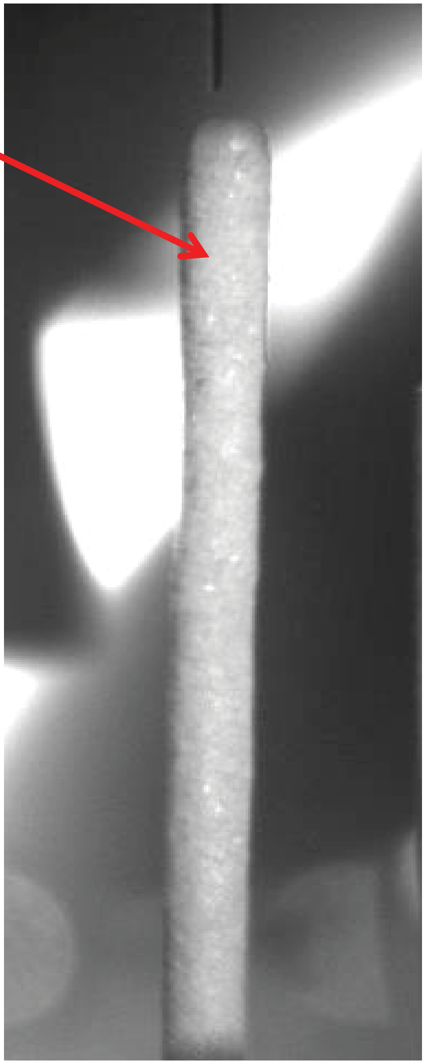
Powders

- Fe(3N5), Si(2N), graphite (3N)
- -325 mesh or < 44 μm in dia.

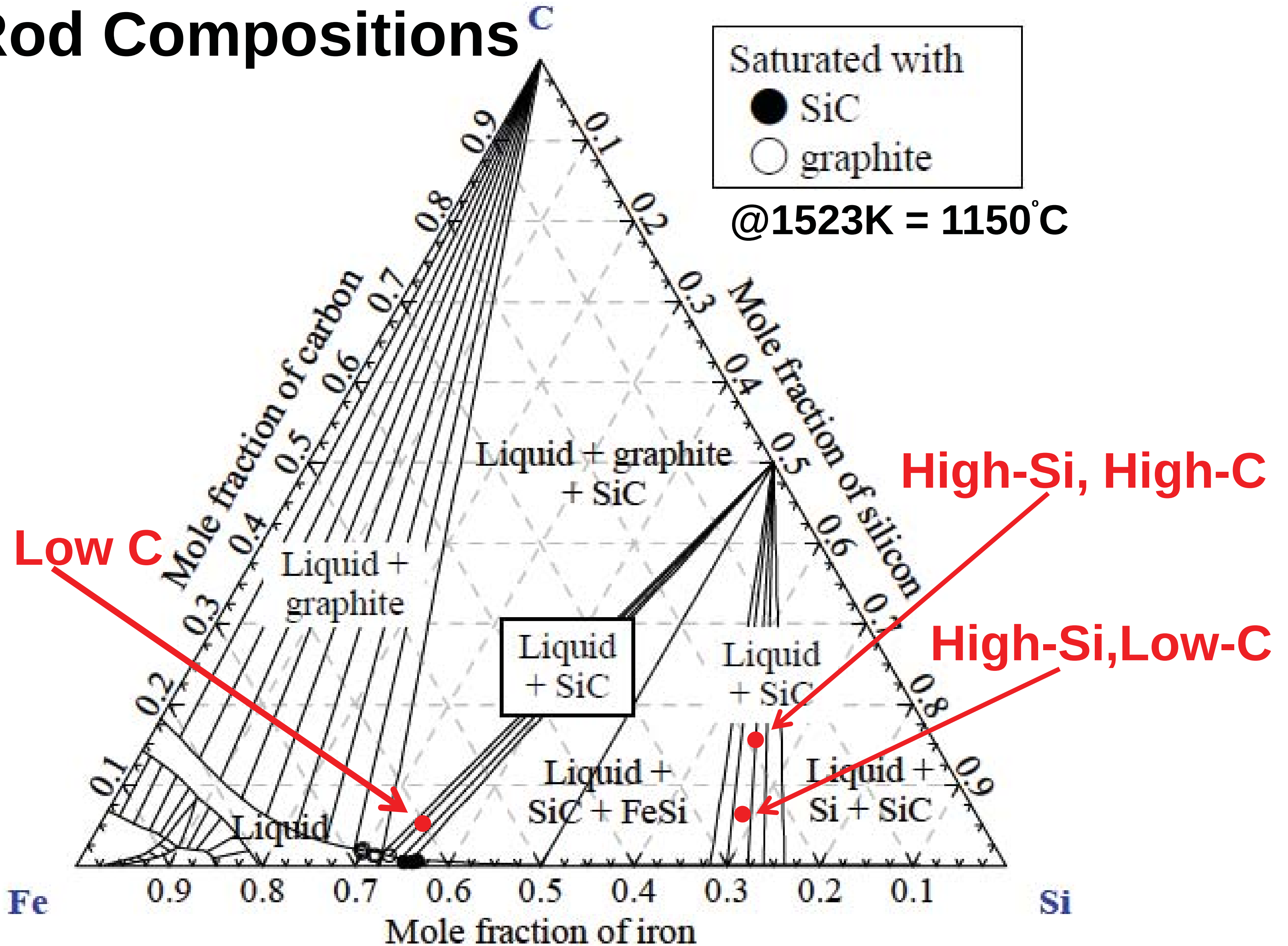
Feed Rod Processing

- Powders mixed by ball mill
- Formed into rods by cold isostatic press
- Sintered @ 1150ºC,1 hour in hydrogen

2 mm dia.



Feed Rod Compositions



"Fundamental study for solvent growth of silicon carbide utilizing Fe-Si melt", T Yoshikawa, S Kawanishi and T Tanaka, *International Conference on Advanced Structural and Functional Materials Design 2008,* Journal of Physics: Conference Series **165** (2009) 012022

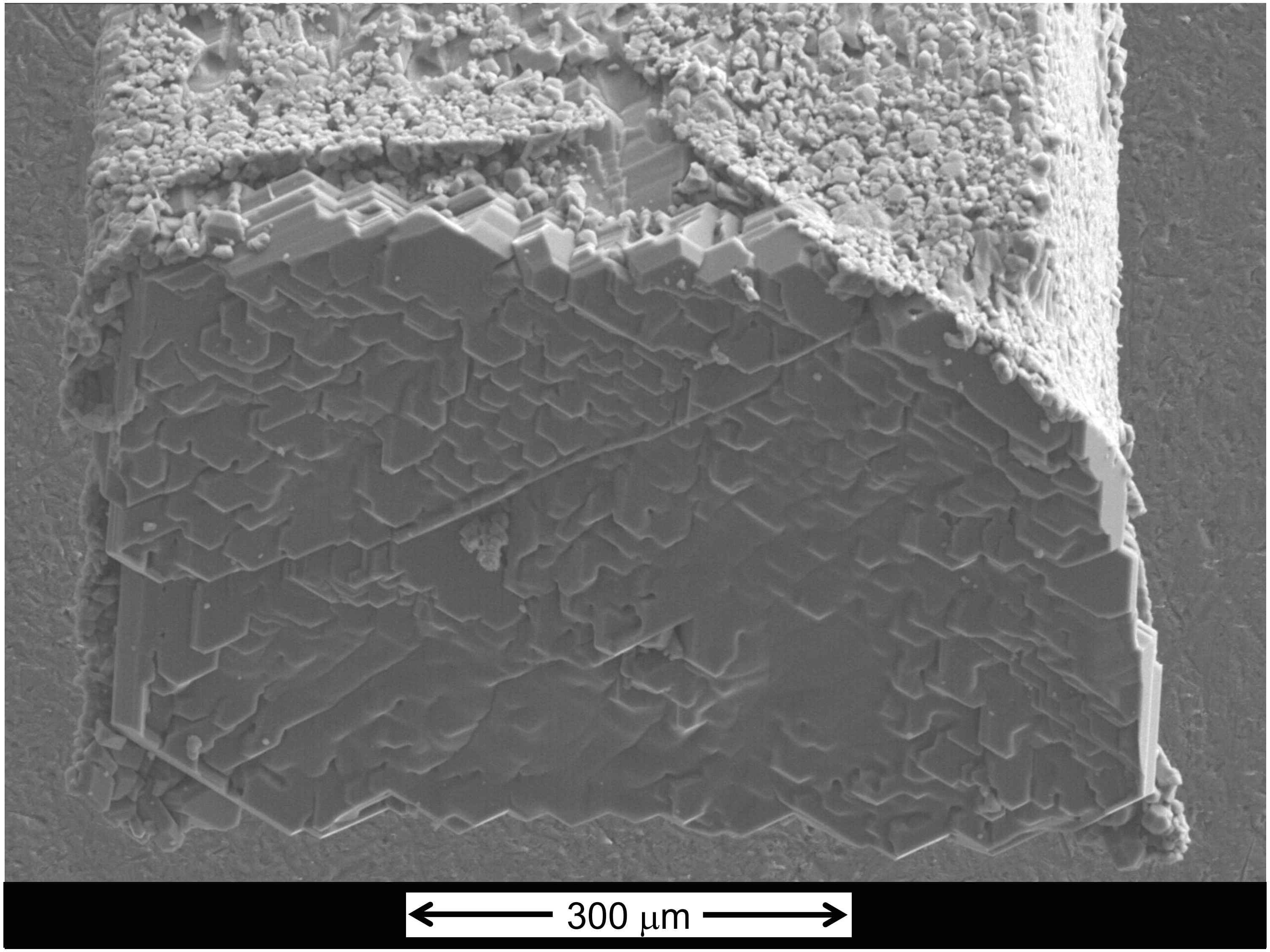
Summary of Results

- X-ray transmission Laue diffraction patterns of the grown crystals
  - Single crystal
  - Retains the 4H-SiC polytype of the seed crystal
- Synchrotron White Beam X-ray Topography
  - Significant inhomogeneous strain

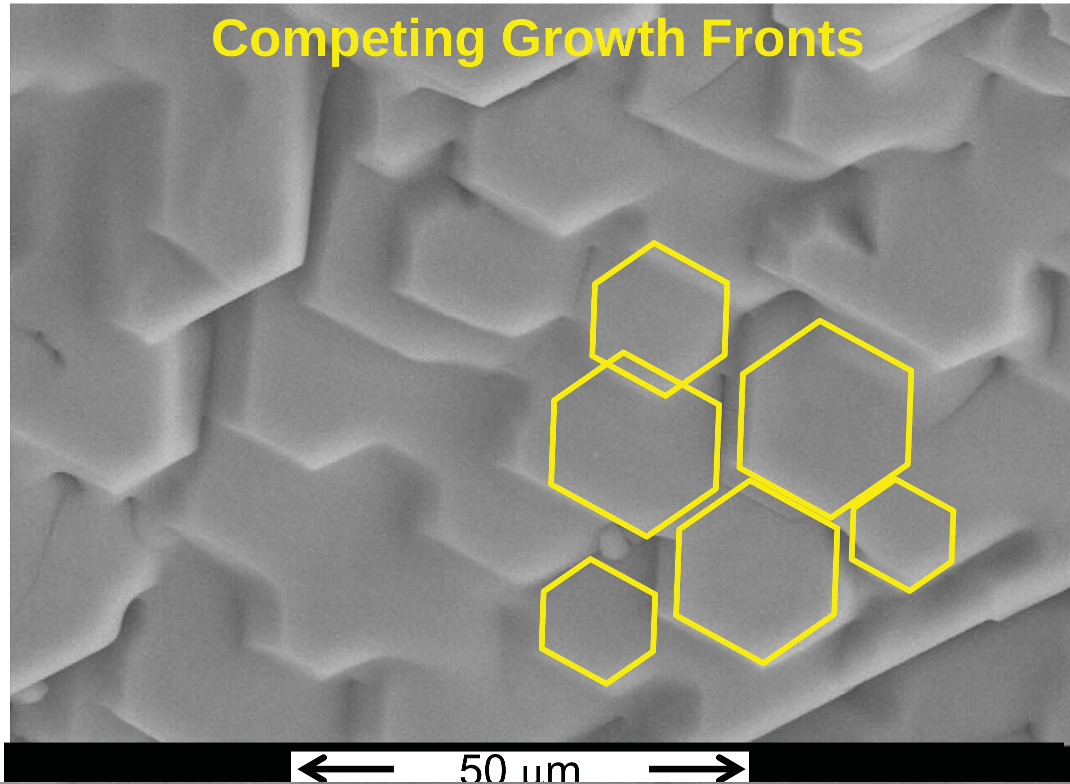
			Growth Rates (µm/hour)/ Fe Concentration (atom/cm^3)		
Fe/Si (atomic ratio)	C (at.%)	M.P. (°C)	M.P.+90 °C	M.P.+190 °C	M.P.+325 °C
High-Si (Fe/Si~0.35)	8	1170	4 / ~10^{17}	40 / ~10^{17}	135
	16	1195	50 / ~10^{18}	120 / ~10^{18}	N/A
High-Fe (Fe/Si~1.9)	8	N/A	No Growth		

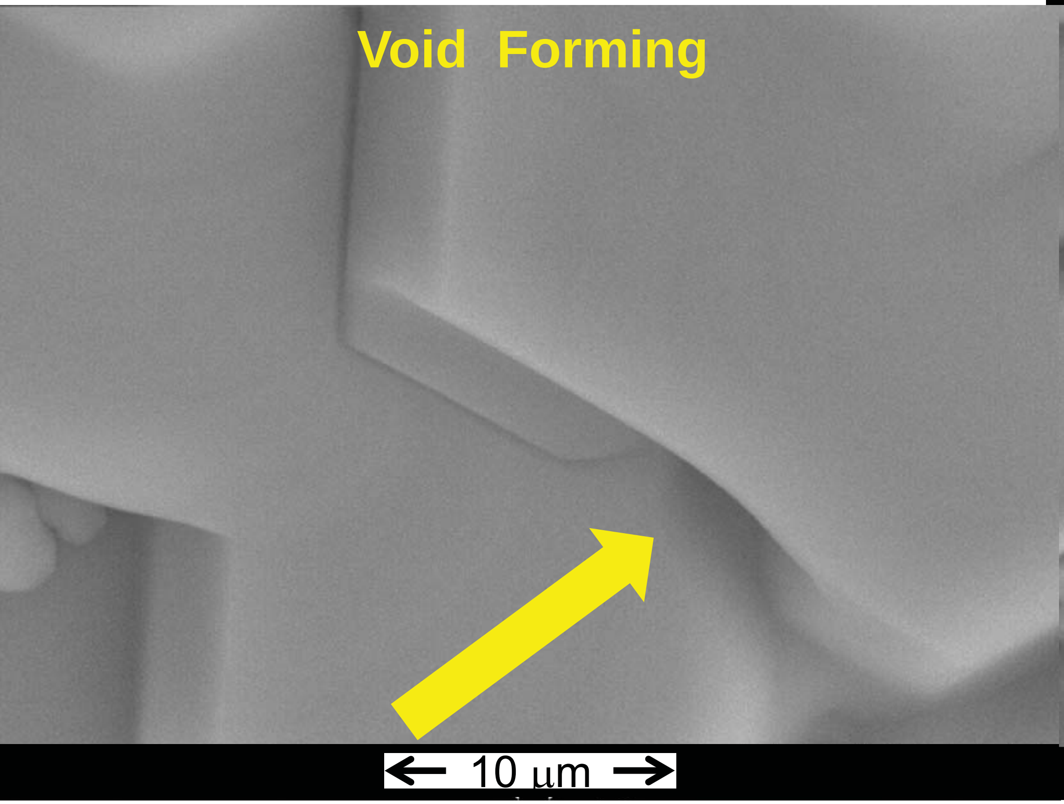
- M.P.= temperature at which the feed rod formed a melt
- at.% =atomic
- Temperatures are not corrected for emissivity

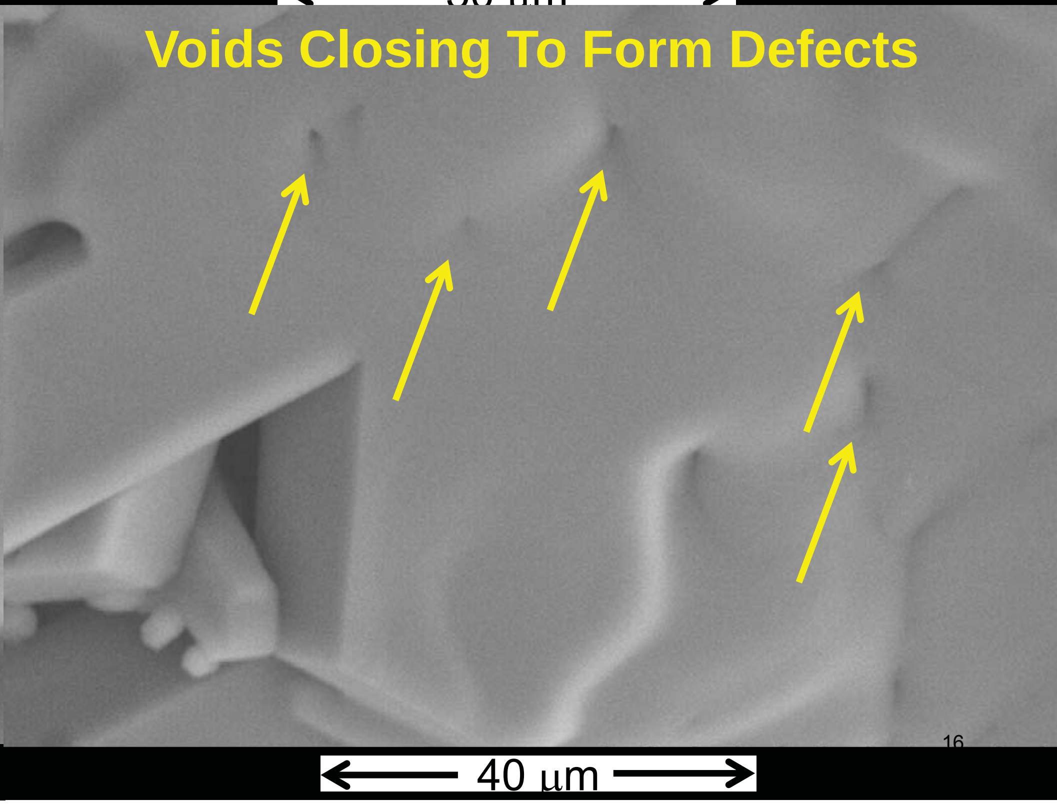
Growth Front Evolution



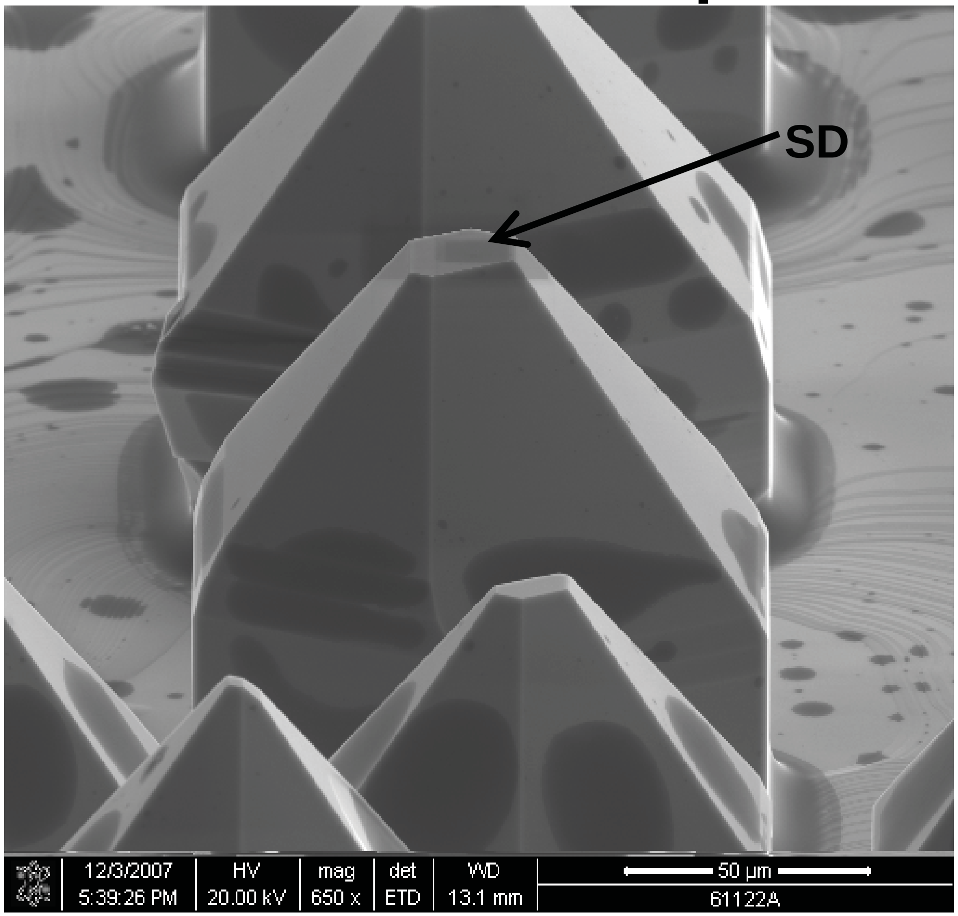
Growth Front Evolution (cont.)

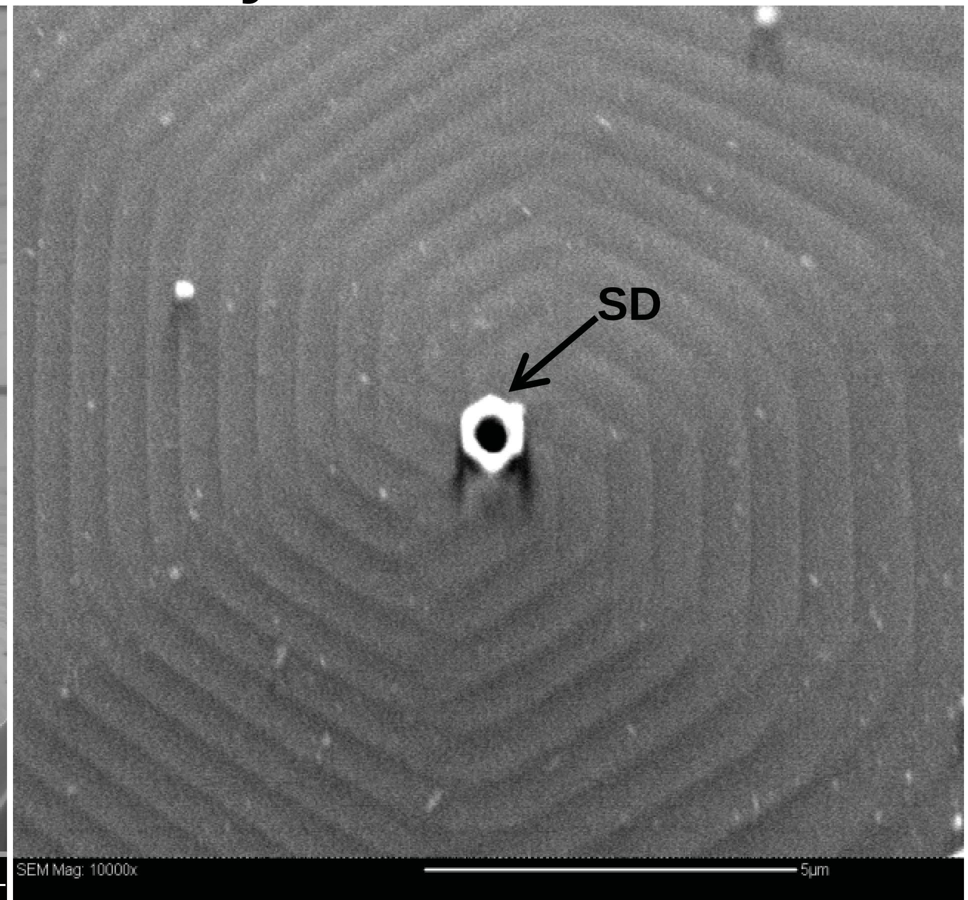






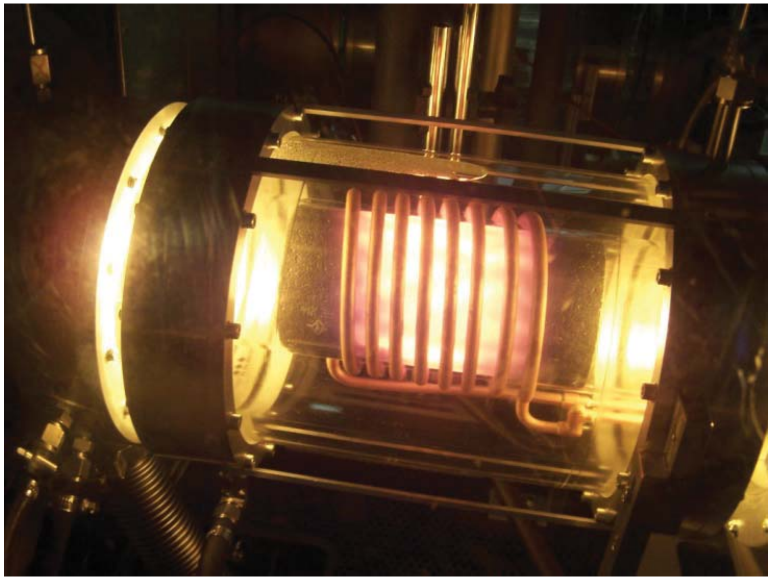
Proposed Seed Crystal



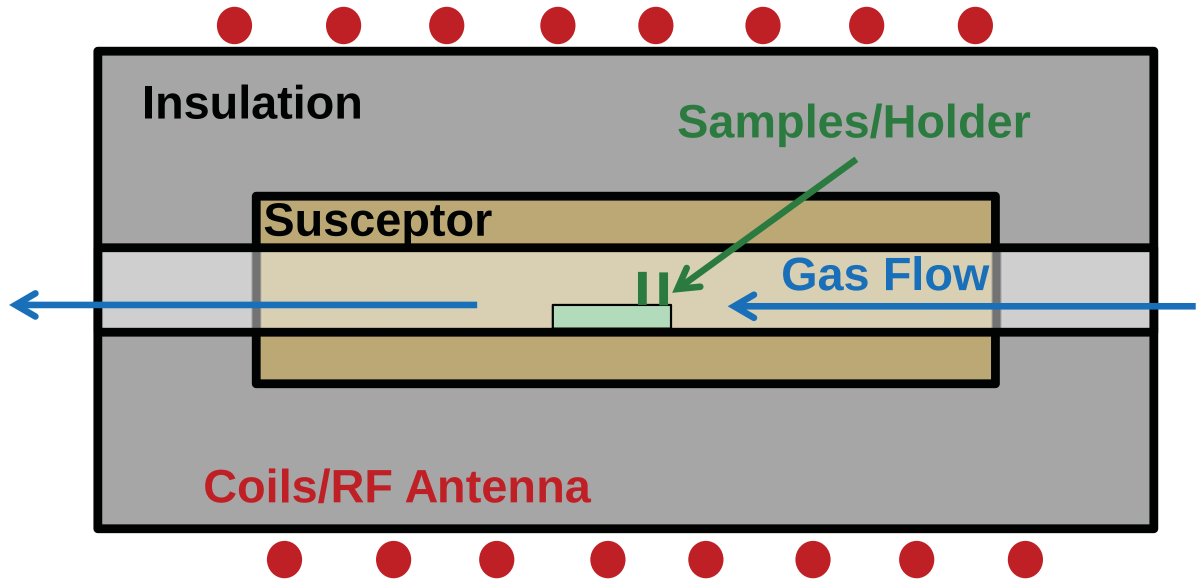


Y. Picard *et al.*, MRS Symp. Proc. Vol. 1069, p. 151 (2008)

Lateral Chemical Vapor Deposition (CVD) Epi-Growth



Cross Section of Custom Hot-Wall Reactor

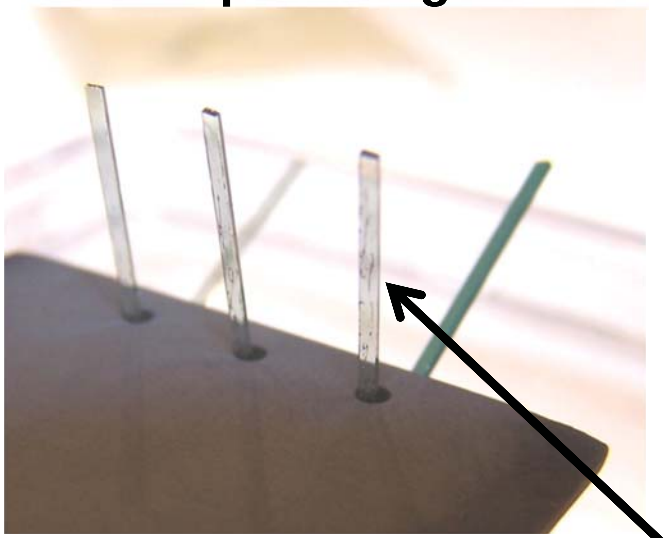


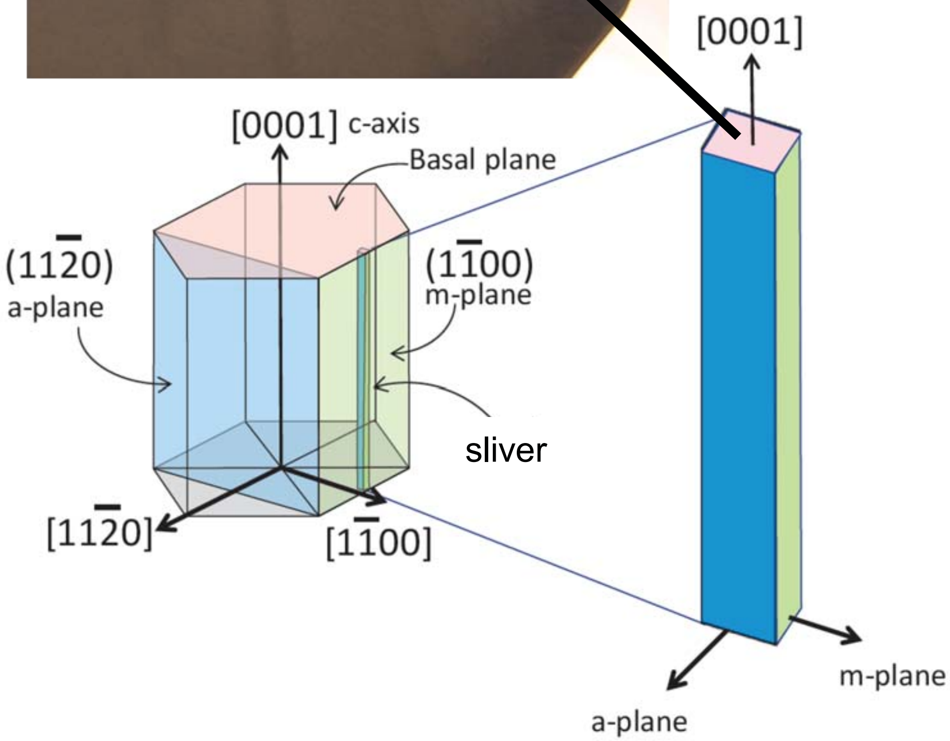
Growth Time [hours]	*In-situ* etch [min]	Etch Pressure [mb]	Growth Pressure [mb]	Hydrogen [sccm]	Silane[1] [sccm]	Propane[1] [sccm]	HCl[1] [sccm]	Estimated Temperature[2] [°C]
5	12	40	325	4260	0/4	1.5/1.5	15/20	1600
16.5[3]	6[3]	40	325	4910	0/8	1.5/2.5	15/40	1600

[1]Etching conditions / growth conditions
[2]Direct observation of temperature by pyrometry was possible. An inferred temperature was calculated based upon melting points Si and Pd
[3] Growth performed in four stages (0.5 ,4 ,4 and 8 hours), insitu etch performed in first stage only.

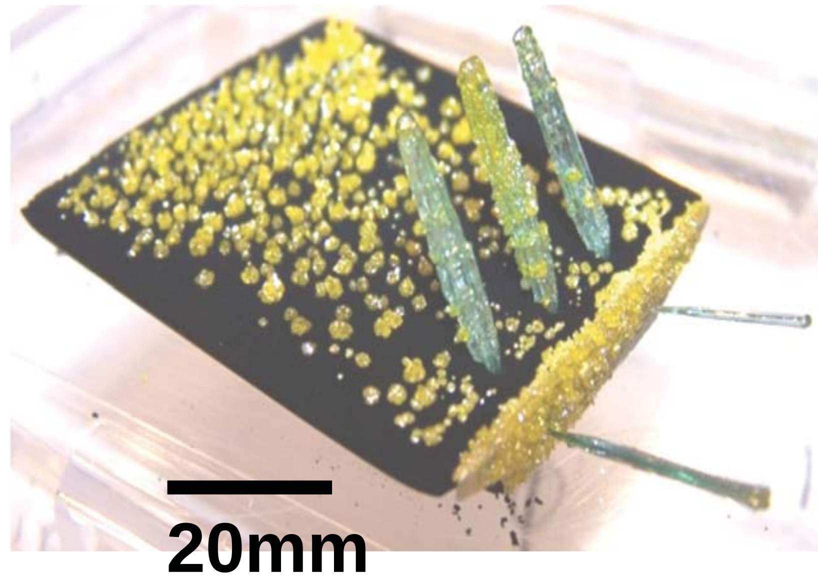
Lateral CVD Epi-Growth 5 Hour Growth

4H/6H SiC a/m-plane slivers prior to growth

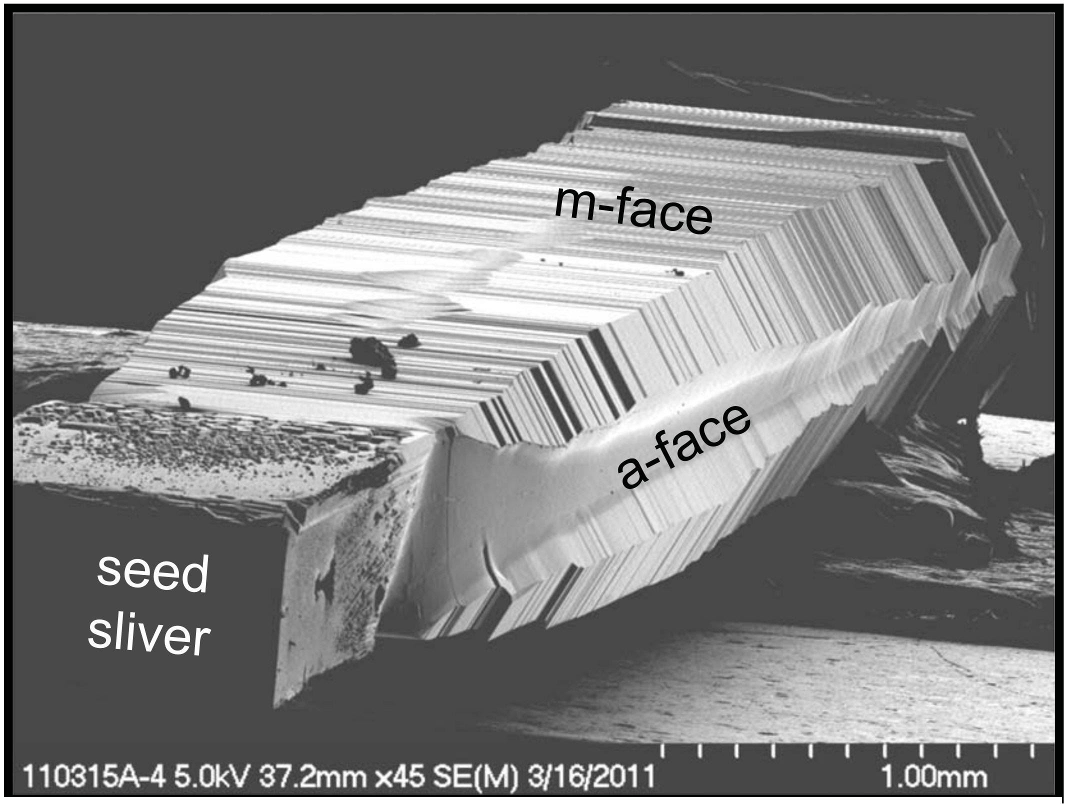




4H/6H SiC a/m-plane slivers post growth

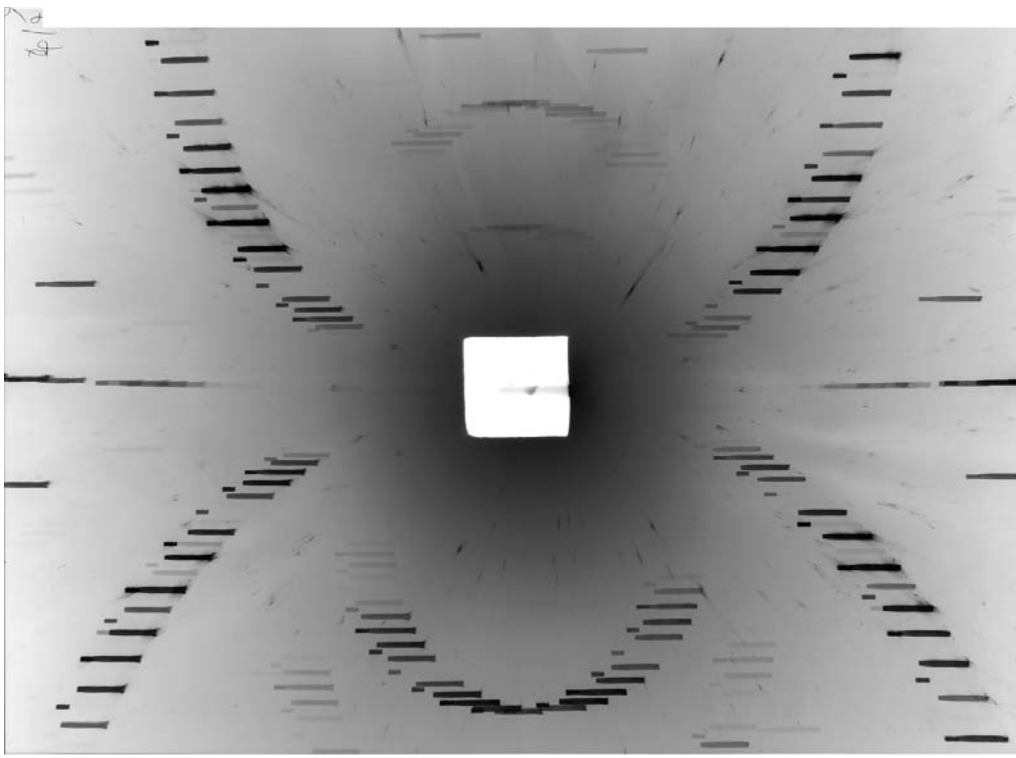


Lateral CVD Epi-Growth 5 Hour Growth (cont.)



Epi Growth Rate: ~80 µm/hour
Max. Film Thickness: ~0.15 mm
Max Diameter: ~1 mm (mostly seed)
Rough grown surfaces/mini-facets

X-ray Topographic Image of Lateral CVD Epi Growth



Grown Crystal

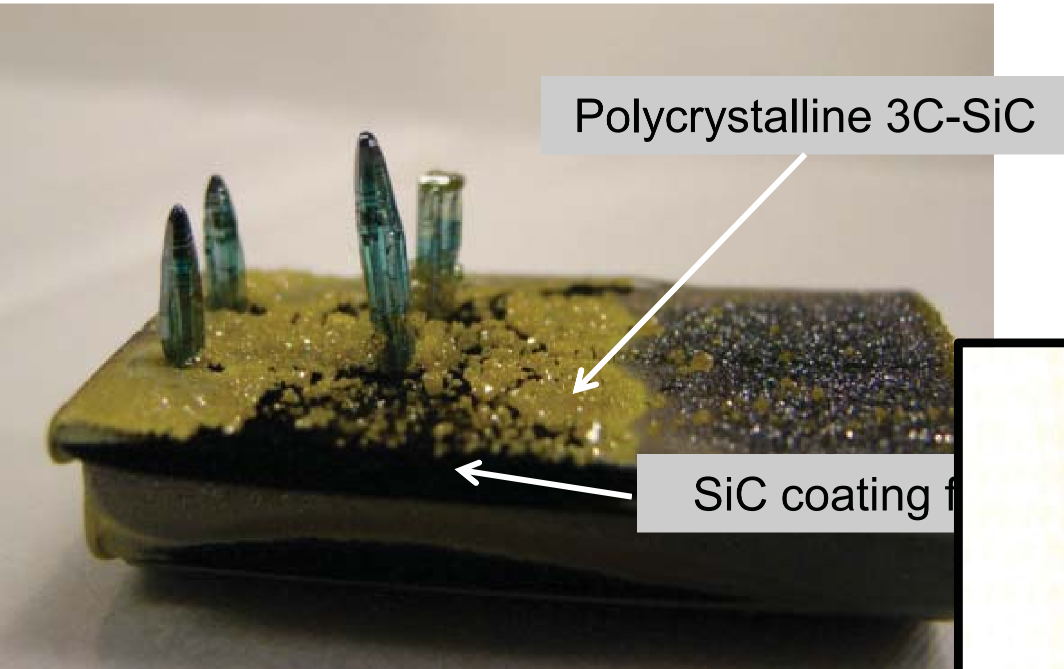
Simulated* 4H-SiC (1-100)

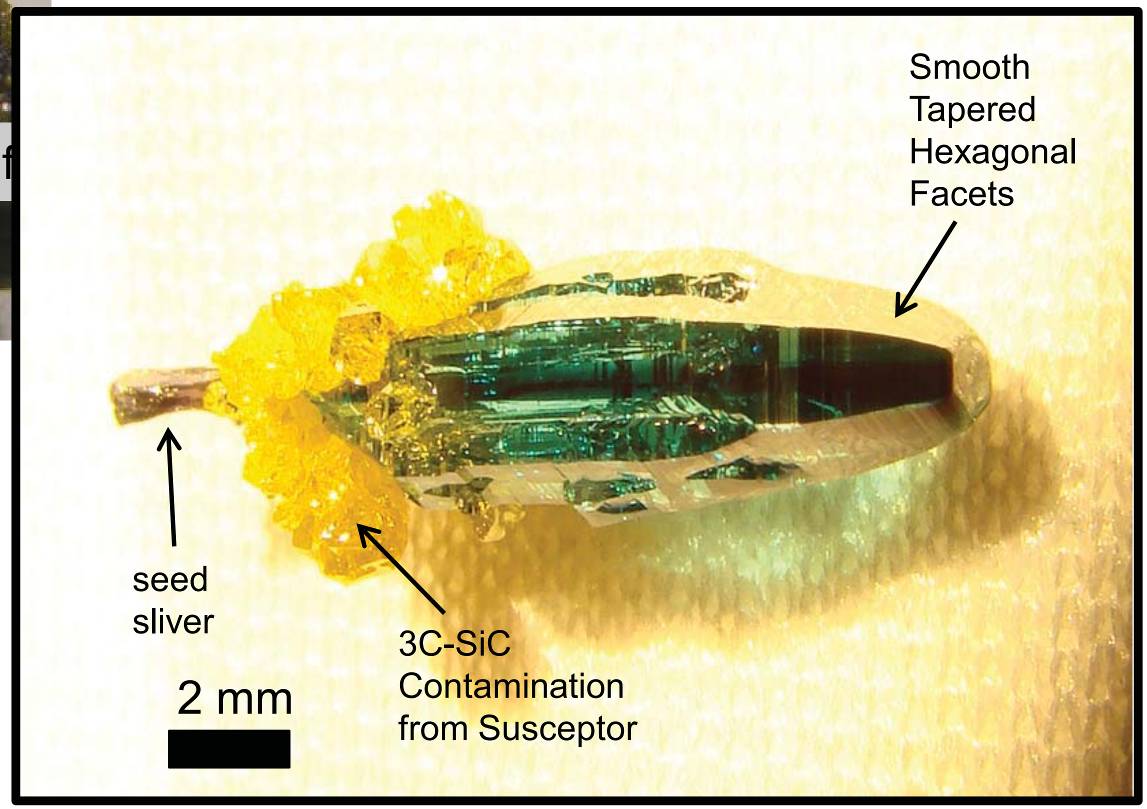
- Courtesy of Balaji Raghothamachar and Michael Dudley
- Recorded at Stony Brook Synchrotron Topography Station, Beamline X19C at the National Synchrotron Light Source, Brookhaven National Laboratory
- *X. R. Huang, J. Appl. Cryst. (2010). 43, 926–928.

X-ray Topographic Image of Lateral CVD Epi Growth

- X-ray transmission Laue diffraction patterns of the grown crystals
    - Single crystal
    - Retains the 4H-SiC polytype of the seed crystal
- Synchrotron White Beam X-ray Topography
    - No long grain strain
    - Some local areas of strain

Lateral CVD Epi-Growth (16.5 hour of growth)





Epi Growth Rate: ~ 120 μm/hour
Max. Film Thickness: ~2 mm
Max Diameter: ~4 mm (mostly epi)
Smooth Tapered Hexagonal Facets!

Conclusions

- Solvent-LHFZ
  - Have grown single crystal SiC
  - Growth Rates in excess of 120 μm/hour
  - Growth fronts are “complex” and therefore create inhomogeneous strain
- Laterial CVD Epi-Growth
  - Growth rates in excess of 120 μm/hour
  - Growth conditions do not seem to be creating crystal defects, but more analysis is needed.

Future Work

- Solvent-LHFZ
  - Implement new seed crystal
  - Continued refinement of source material/ feed rods
- Laterial CVD Epi-Growth
  - Extend growth of boule beyond 5mm
  - Confirm CVD growth is not inducing new defects

Areas for Collaboration

- Start a parallel effort in GaN
- Alternative uses for SiC fibers (unique structure)
- Lateral growth on SiC fibers may be able to create other unique structures

Team Members

RHS
(SiC growth, sensors & electronics)
Phil Neudeck
Andy Trunek
David Spry
Tony Powell (retired)
Michelle Mrdonovich-Hill
Beth Osborn
Chuck Blaha

RXC
(Ceramics)
Ali Sayir
Fred Dynys
Thomas Sabo

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NASA